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Optimizing the Oil Extraction/Water Adsorption Step in Sequential Extraction Processing of Corn

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ABSTRACT. *The Sequential Extraction Process (SEP) uses ethanol to extract oil and protein from cracked, flaked, and dried corn, and the corn simultaneously dehydrates the ethanol. The optimum conditions to reduce zein extraction in the oil extraction/water adsorption step of SEP involved a single–pass system using 15 length/diameter ratio extraction cells, 30% hexanes:70% ethanol, and 56°C extraction temperature, which improved the efficiencies of oil extraction from the corn and moisture adsorption from the ethanol. The extract contained only 0.1% protein in the desolventized solids. The moisture content of the solvent was reduced to 1.03%, and the residual oil content of the corn was 0.18%. Oil recovery was improved from 90.8% to 95.5%. The countercurrent laboratory extraction system confirmed that using 30% hexanes:70% ethanol at 56°C in a single–pass system reduced zein co–extraction with oil while increasing oil extraction and moisture adsorption.*

Keywords. *Corn, Ethanol, Extraction, Maize, Milling, Zein, Corn oil, Corn protein, Corn processing.*

The Sequential Extraction Process (SEP) (fig. 1) is a multi–step extraction process in which ethanol produced from fermenting cornstarch is used to extract oil and protein from cracked, flaked, and dried corn (Hojilla–Evangelista et al., 1992a, 1992b). Several value–added co–products are possible when producing fuel ethanol, potentially making ethanol production more economical (Johnson et al., 1994; Chang et al., 1995). In the oil extraction step of SEP, water is also simultaneously adsorbed from the ethanol by the dried, flaked corn to produce 99% ethanol (Robertson and Pavlath, 1986; Chen and Hoff, 1987; Chein et al., 1988). The moisture–adsorption capacity of soft dent corn dried to less than 2% moisture was found to be 22 g water/kg corn (Hojilla–Evangelista et al., 1992b). A small amount of protein, identified as predominantly zein, is also co–extracted with the oil. The miscella (solvent plus extractants) is evaporated to yield ethanol and a mixture of crude oil and zein. The zein must be separated from the oil by washing with small amounts of hexane and filtering. In a subsequent protein extraction step, a food–grade protein concentrate (>70% protein dry basis) is extracted with a mixture of ethanol and alkali.

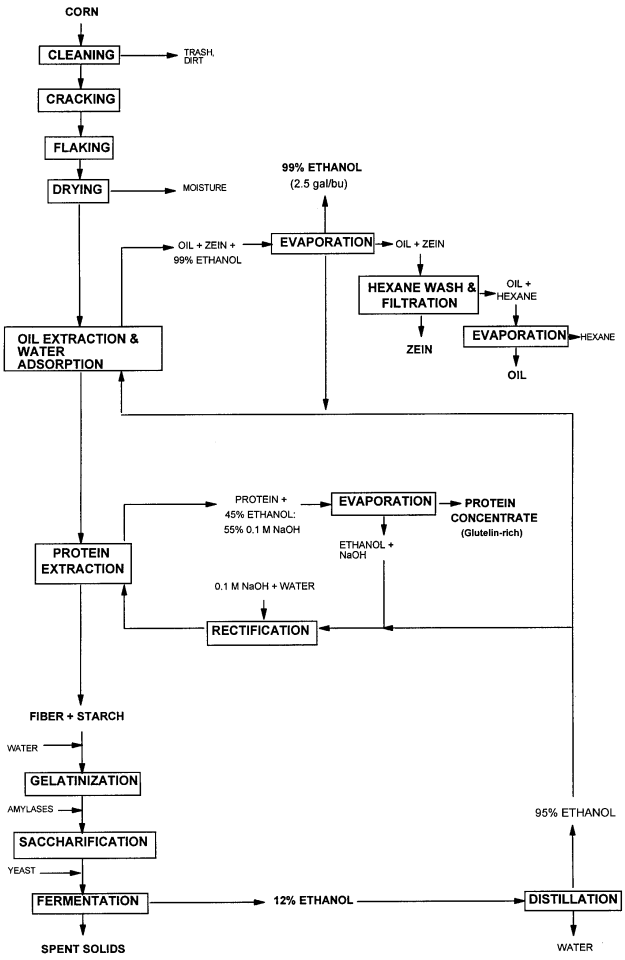

Figure 1. The original Sequential Extraction Process for corn.

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Unlike most corn protein products produced to date, SEP protein concentrate has high water solubility, good foaming properties, high heat stability, and good emulsification capacity and stability (Hojilla–Evangelista et al., 1992c, 1996). The fiber and starch fraction that remains is gelatinized, saccharified, and fermented to produce ethanol. The dilute ethanol beer (approximately 12%) is distilled to 95% ethanol and is recycled upstream to be used in the protein extraction step and the water adsorption/oil extraction step before exiting the plant to be sold for blending with gasoline.

In the present process design, zein recovery and purification are difficult because of the presence of large quantities of oil. It seemed preferable to shift zein extraction downstream by employing a blend of non–polar solvent and ethanol for the oil extraction/water adsorption step. This solvent would have polarity that is proportional to the amounts of ethanol and non–polar solvent mixed together. The solvent may be sufficiently less polar than ethanol alone to reduce the amount of zein co–extracted and to improve the quality of oil because of reduced amounts of extracted polar lipids (Feng et al., 2000). Hexane and unleaded gasoline are logical choices as polarity–adjusting solvents (PAS). Gasoline has been used for oil extraction (Johnson and Lusas, 1983) and as a denaturant for fuel ethanol.

Youngquist (1976) patented a process that used a hexane/ethanol/water mixture to deflavor seed proteins. This solvent mixture forms an azeotrope (78.5 hexane:20.8 ethanol: 0.7 water) in a gaseous phase at 58.6°C. Upon vapor condensation, however, the azeotrope splits into two phases, with the upper phase having a weight ratio of 96.5 hexane: 3.0 ethanol:0.5 water and the lower phase having a weight ratio of 6:75:19.

After oil extraction using ethanol and PAS, a two–step protein extraction method could then be used to maximize zein recovery and purity. In this option, a separate zein extraction step using 80% ethanol would be added between the oil extraction/water adsorption step and the glutelin extraction step.

The principal research objective of these experiments was to determine the optimum conditions to reduce zein extraction in the oil extraction/water adsorption step while maintaining satisfactory oil extraction from the corn and moisture adsorption from the ethanol. We hypothesized that: (a) oil extraction would be maintained or improved by the addition of non–polar solvent to ethanol; (b) protein extraction would be reduced because of decreased polarity of solvent; and (c) water adsorption from the solvent would be enhanced because the decreased polarity would make the solvent less compatible with water, and the water would be more easily adsorbed by the flaked corn.

Materials and Methods

Corn Preparation

Batches of soft dent corn (Pioneer 3377, Pioneer Hi–Bred International, Inc., Johnston, Iowa), each weighing 350 g, were cracked and then flaked to 0.5 mm (0.02 in) thickness by using a Roskamp rollermill (Model K, Roskamp Mfg., Inc., Waterloo, Iowa). The flakes were dried at 55°C in a forced–air convection oven to a moisture content of 1.12% (weight basis, wb) for use in SEP simulations. Each batch was individually sealed in a polyethylene bag and placed in a desiccator at ambient temperature until used. Moisture content of each corn batch was determined by Karl Fischer titration (ASTM, 1986).

Single–Stage Extraction and Adsorption Trials

Single–stage extraction trials were conducted to determine the effects of temperature and level of PAS on extraction rates of oil and protein from corn and on moisture adsorption from the solvent. The control solvent was 95.5% (wb) ethanol, added at 76°C to the dried, flaked corn at a 2:1 solvent–to–corn (S/C) ratio. These conditions corresponded to those of incoming ethanol used in previous studies (Hojilla–Evangelista et al., 1992b, 1992c). For the ethanol/PAS (unleaded gasoline or hexane) solvent mixtures, the extraction temperature was reduced to 56°C because of the lower boiling points of their azeotropes (Johnson and Lusas, 1983). Hexane or unleaded gasoline was added to ethanol at 5%, 10%, and 30% (wb) of the total solvent mixture. Extraction using aqueous ethanol at 56°C was also done for comparisons with results obtained at 76°C and by adding PAS. Duplicate extractions were done for each treatment. The ethanol–PAS mixture that gave optimum results was used in the next set of extraction trials.

The batch extraction system (fig. 2) used jacketed glass columns (9.5 cm i.d. × 20 cm length) for holding the corn and solvent. The extraction temperature was regulated by circulating water from a hot–water bath through the columns and monitored by thermometers inserted through the stoppers that covered the columns. Solvent evaporation was minimized by using cold–water condensers. Entry of atmospheric moisture was prevented by drying tubes attached to condenser vents and by flushing the system with dry nitrogen gas.

Extraction was started when the system (including the solvent) attained the appropriate temperature. The solvent was pumped through the heat exchanger by a diaphragm pump (Masterflex Model 7090–42, Cole–Parmer Co., Chicago, Ill.) and allowed to percolate repeatedly through the flaked corn bed for 70 min, simulating the total contact time between solvent and corn in the countercurrent system (Hojilla–Evangelista et al., 1992b, 1992c). Ten–milliliter aliquots of the circulating solvent were taken at 0, 2, 4, 6, 8, 10, 30, and 70 min. These aliquots were analyzed for moisture (ASTM, 1986), crude lipid (AACC, 1983), and crude protein contents (AACC, 1983).

Breakthrough Adsorption Curves

These trials were performed to determine the effects of solvent composition and extraction column geometry (which affects the length–to–diameter [L/D] ratio of the column and the number of solvent passes through the flake bed over a given time period) on oil, protein, and moisture transfers. The solvents were 95.5% (wb) ethanol at 76°C (control) and 95.5% ethanol:hexane mixture (7:3 ratio) at 56°C. Each solvent was tested on two corn extraction columns: the original column (9.5 cm i.d. × 20 cm length, L/D ratio of 2), and the modified column (4 cm i.d. × 63 cm length, L/D ratio of about 15). The single–stage batch extraction system was also used for these trials. Solvents were added to the dried, flaked corn by using a 5:1 S/C ratio. A diaphragm pump maintained the flow rate at 50 mL/min. The effluent was collected in 52–mL aliquots, which were analyzed for

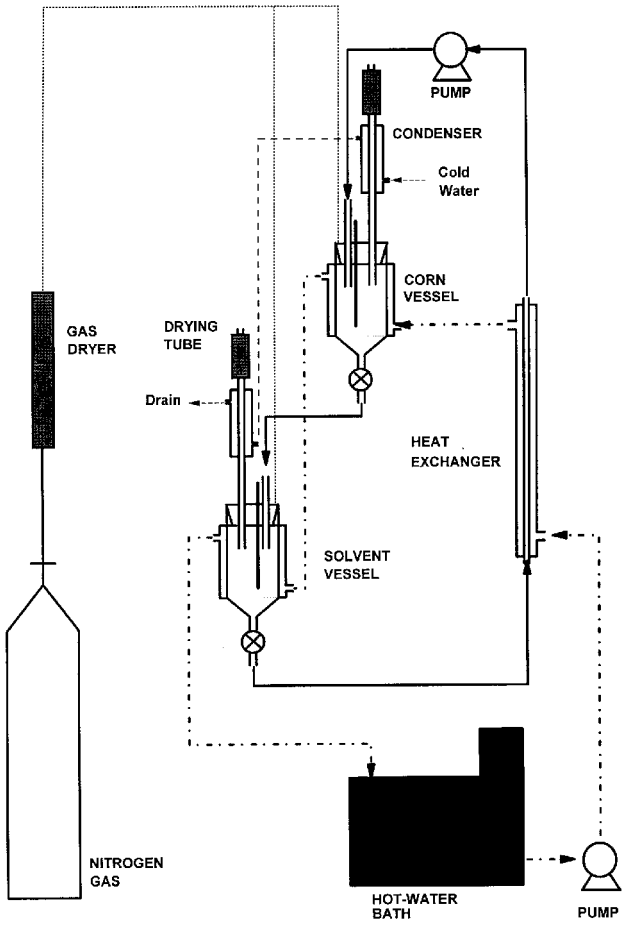

Figure 2. Single–stage batch extraction system.

moisture (ASTM, 1986), crude lipid (AACC, 1983), and crude protein contents (1983). Duplicate extractions were done for each treatment.

Countercurrent Oil Extraction and Moisture Adsorption

The countercurrent oil extraction/moisture adsorption system to simulate SEP was set up as described by Hojilla–Evangelista et al. (1992b), except that the solvent was reconstituted instead of reclaimed from the rotary evaporator. The starting miscellas were prepared as shown in table 1 and are presented in order of solvent contact with the corn (columns 1–7). The water content of miscella 7 (incoming solvent) was determined by mass balance and corresponds to 2.5 gal of 95% ethanol per bushel (370 L/metric ton) of corn, as is produced in practice. The other water contents were determined by approximating an exponential solution curve, as was done by Hojilla–Evangelista et al. (1992b). Hexanes constituted 30% (wb) of the solvent fraction, and ethanol constituted the other 70%.

Table 1. Starting miscella compositions (% wb).

	Miscella						
	1	2	3	4	5	6	7
Organic solvent	99.4	99.4	99.4	99.2	99.0	98.6	97.7
Ethanol	69.6	69.6	69.6	69.5	69.3	69.0	68.4
Hexanes	29.8	29.8	29.8	29.7	29.7	29.6	29.3
Water	0.6	0.6	0.6	0.8	1.0	1.4	2.3

The two treatments used in the countercurrent adsorption/extraction system are shown in table 2. Each treatment was replicated twice. The control was the original system, as described by Hojilla–Evangelista et al. (1992b), using aqueous ethanol at 76°C. This treatment was not replicated in this work because the corn and all other parameters were the same as previously used (Hojilla–Evangelista et al., 1992b). The results for the multi–pass 30% hexanes and the single–pass 30% hexanes were compared with these previously published data. When the short extraction column (L/D ratio of 2) was used in the SEP system, the solvent was recycled through the flake bed for 10 min and drained for 5 min. When the modified extraction column (L/D ratio of 15) was used, solvent flow through the flaked corn was changed from multiple passes to single pass. Solvent flow was slower in the longer column and thus did not require multiple passes. The new flow rate was determined by gravity flow through the flake bed. Both systems were controlled to provide 10 min of contact time between solvent and corn per extraction stage. Steady–state was assumed when moisture contents of the recovered solvent and marc (solvent–laden corn solids), solvent holdup levels, and oil yields remained constant.

Analysis of Samples

Both liquid and solid samples were tested for moisture content by using the Karl Fischer titrimeter system (ASTM, 1986). The crude lipid contents of liquid samples were determined by extracting volatile–free samples with hexane and filtering through Whatman No. 54 hardened filter paper. The hexane extracts were evaporated, dried, and weighed to determine amounts of lipid. Crude lipid contents of solid samples were analyzed by using AACC method 30–20 (AACC, 1983).

The micro–Kjeldahl method (AACC, 1983) was used to estimate protein contents in liquid samples. Samples were reheated to the extraction temperature and mixed before measuring samples into Kjeldahl flasks for analysis. Rotary–evaporated samples were defatted by extracting with two 40–mL aliquots of chilled hexane (to reduce any possible protein extraction) and removing the oil–rich hexane layer. Samples were again rotary–evaporated to dryness to remove hexane. Standard micro–Kjeldahl procedures (AACC, 1983) were used for the remainder of the procedure, except that the concentration of the HCl was reduced to 0.01 N to accommodate the low levels of protein. A nitrogen conversion factor of 6.25 was used to calculate crude protein contents.

Table 2. Treatments for countercurrent adsorption and extraction curves.

Parameters	Control	Multi–pass system, 30% hexanes	Single–pass system, 30% hexanes
System change	None	Solvent	Solvent and column geometry
Solvent(s)	100% ethanol	30% hexanes, 70% ethanol	30% hexanes, 70% ethanol
Extraction temperature	76°C	56°C	56°C
Column L/D ratio	2	2	15
Solvent flow	Recirculated	Recirculated	Single–pass

Crude protein contents of the solid samples were estimated by using AACC standard method 46–08 (AACC, 1983), except that copper (II) selenite dihydrate was used as catalyst. A nitrogen conversion factor of 6.25 was used.

Statistical Analysis

Statistical analysis was performed by using Statistica software. Multiple ANOVA and Duncan's Multiple Range tests were performed on steady–state data to determine differences between the treatments.

Results and Discussion

Effects of Extraction Temperature and Polarity–Adjusting Solvents

Moisture transfer from the solvent to the dried, flaked corn was indicated by the reduced moisture content of the eluted miscella. In all treatments, moisture transfer was observed to be almost completed within 10 min. Moisture adsorption by corn was greater at 56°C than at 76°C (fig. 3). At 56°C, moisture adsorption capacity (MAC) was 4.4 g water/100 g corn, while at 76°C, MAC was 2.8 g water/100 g corn. At the lower extraction temperature, the energy state is lower and water molecules have less mobility because the hydrogen bonds between water molecules and adsorbent (corn) are not as easily destroyed. The net effect is a stronger tendency to remain bonded to the adsorbent instead of migrating to the aqueous phase. Corn adsorbed more water from the solvent mixture that contained 30% hexanes (MAC = 3.7 g water/100 g corn) and the least amount of water from the control solvent (95.5% (wb) ethanol, designated as 0% PAS, at 76°C) (fig. 3). This behavior affirmed our hypothesis that water adsorption from the solvent should be enhanced by adding PAS, because the decreased polarity should make the solvent less compatible with water and the water should be more easily adsorbed by the flaked corn.

Temperature had a much greater effect than level of PAS on protein extraction. Reducing the extraction temperature from 76° to 56°C without adding PAS reduced the amount of protein co–extracted with oil to about 40% of the amount obtained from the original system (fig. 4). Addition of hexanes further reduced the amount of co–extracted protein. Less protein was extracted at higher levels of hexane. The amount of protein extracted by the blend of 30% hexanes:70% ethanol was less than 10% of that extracted by the control solvent (0% PAS, 76°C). Unleaded gasoline was less effective than hexane in reducing protein extraction.

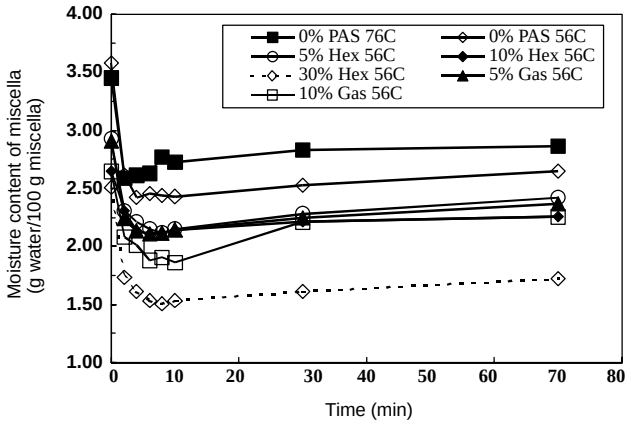

Figure 3. Effects of temperature and PAS on water adsorption by corn.

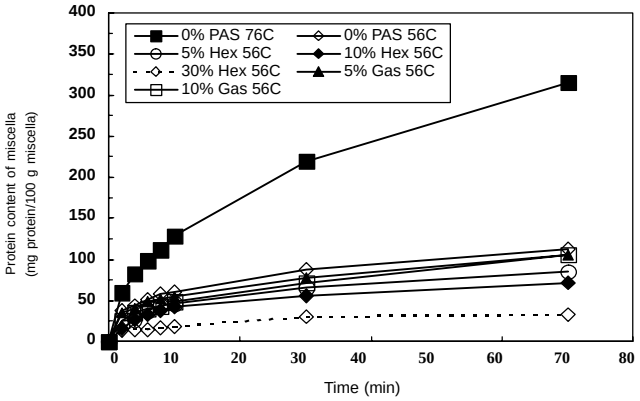

Figure 4. Effects of temperature and PAS on protein extraction.

Reducing the extraction temperature did not reduce the amount of oil recovered from corn (fig. 5). Addition of unleaded gasoline did not increase oil extraction. Oil recovery increased slightly when 10% and 30% hexane was added to ethanol. In all the treatments, most of the oil was extracted after 30 min.

Effects of Extraction Column Geometry

Breakthrough curves for solvent treatments that used the long, narrow column (15 L/D ratio) exhibited a true breakthrough point, which is the point where the moisture content of the output solvent begins to rise. The longer the miscella moisture level stayed below that of the input solvent, the greater the drying capacity of the corn. Adsorption of water from the solvent by the flaked corn was much more efficient in the long, narrow column than in the short column (fig. 6).

Another indicator of adsorption efficiency is the expected effective solvent ratio, which is the solvent level that would be used in actual operation of SEP and was calculated by subtracting the hold–up from the solvent ratio (e.g., a 2:1 S/C ratio and solvent hold–up of 60% has an effective solvent ratio of 1.4). The greater the area under the moisture curve up to the expected effective solvent ratio line, the greater the efficiency of moisture adsorption. The long column provided a much greater adsorption efficiency than the short column (fig. 7). In the case of the control solvent (aqueous ethanol at 76°C), the amount of water adsorbed by corn increased by

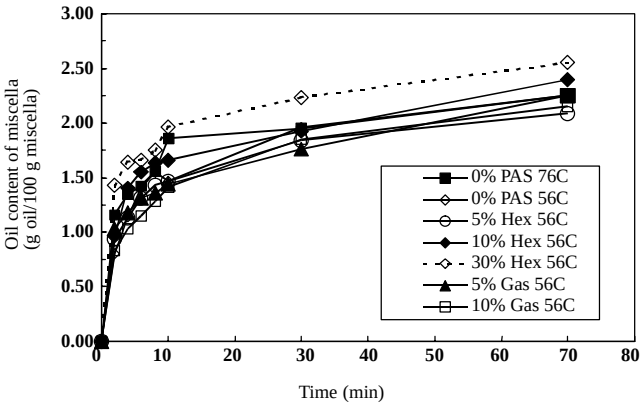

Figure 5. Effects of temperature and PAS on oil extraction.

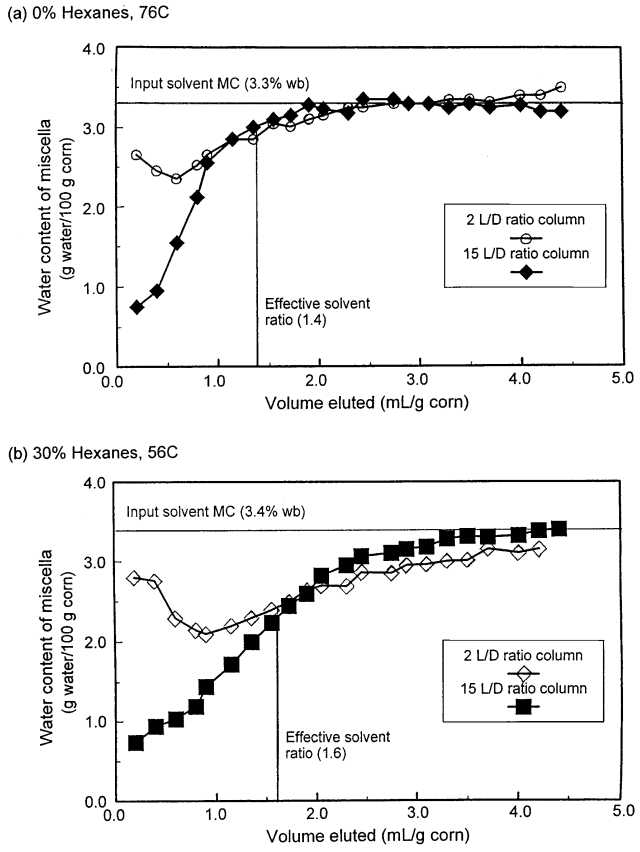

Figure 6. Effects of column geometry on water adsorption: (a) 0% hexanes, 76 C; (b) 30% hexanes, 56 C.

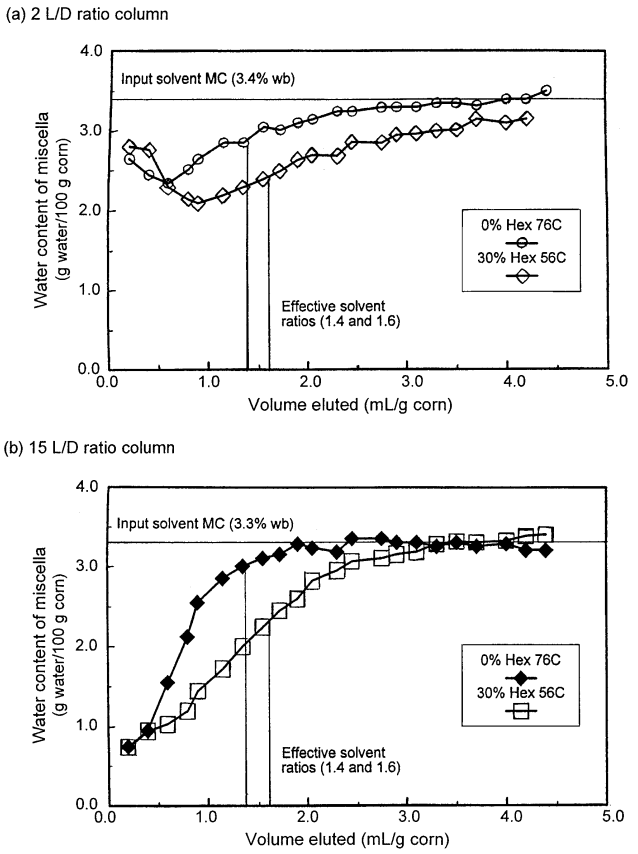

Figure 7. Effects of PAS on water adsorption: (a) 2 L/D ratio column; (b) 15 L/D ratio column.

66% when the long column was used, while for the ethanol:hexane blend, water adsorbed increased by 20%. When the same type of extraction column was used, addition of 30% hexanes to ethanol resulted in greater and more efficient water adsorption (fig. 7), as indicated by the later manifestation of its breakthrough point and the longer time it took for the miscella to approach the moisture level of input solvent.

For both solvents, less protein was extracted with oil when the short column was used (fig. 8). Nearly 50% more protein was extracted by the solvent with the long extraction column. Addition of hexanes dramatically reduced the amount of co–extracted protein in the miscella, especially when the long column was used. Aqueous ethanol extracted more than seven times as much protein as the ethanol:hexanes blend, regardless of column geometry.

Both solvents extracted nearly the same amounts of oil using either extraction column (fig. 9). For each solvent, slightly more oil was extracted by using the long column, but the increase was not significant.

Extraction Using the Laboratory Countercurrent System

Marc moisture levels significantly increased from the starting dry flake moisture levels of 1.12% (table 3), indicating that moisture from the solvent was adsorbed into the corn solids. The recovered solvent moisture content (hexane–free basis) was markedly reduced from its initial moisture content of 2.3% (wb).

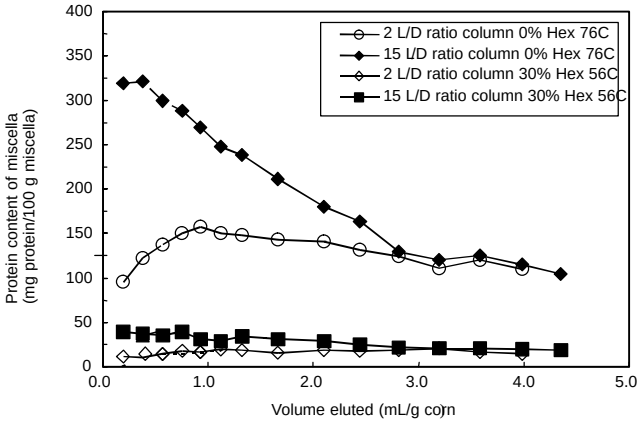

Figure 8. Effects of column geometry and PAS on protein extraction.

The moisture content of the marc from the single–pass 30% hexanes treatment was significantly higher than those from the multi–pass 30% hexanes treatment and the control, indicating better adsorption of water from solvent. The moisture content of the recovered solvent also reflected the same trends (table 3). The enhanced water extraction was attributed to changing the column configuration of the system (larger L/D ratio).

The moisture contents of the exiting miscellas and the recovered solvents were determined for both runs. A discrepancy was noted between the recovered solvent moisture content and the final miscella 1 (full miscella) moisture content after adjusting for the oil contents of the

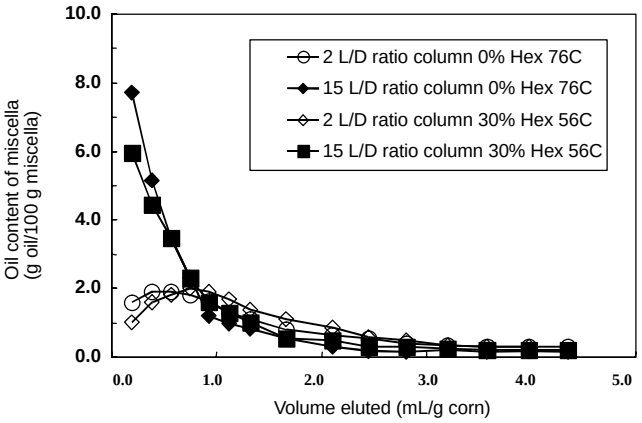

Figure 9. Effects of column geometry and PAS on oil extraction.

Table 3. Moisture contents (MC) of marc and recovered solvent during countercurrent extraction.

Parameters	Control[a]	Multi–pass system, 30% hexanes[b]	Single–pass system, 30% hexanes[b]
System change	None	Solvent	Solvent and column geometry
Marc MC (%, wb)[c]	3.28 ±0.14 b	3.40 ±0.09 b	3.57 ±0.11 a
Corrected recovered solvent MC (% wb)[d]	2.13 ±0.17 a	1.22 ±0.05 b	1.03 ±0.13 c

[a] Means of 6 runs at steady–state.
[b] Means of 7 runs at steady–state.
[c] Means within a row followed by a common letter are not significantly different at $p < 0.05$.
[d] Hexane–free basis. Means within a row followed by a common letter are not significantly different at $p < 0.05$.

miscella. It was concluded that there must have been an air leak in the laboratory SEP system, possibly in the rotary evaporator, so that moisture uptake by the solvent occurred. To calculate a correction value, samples of reconstituted full miscella (miscella number 1) were tested for moisture content, rotary–evaporated under the same conditions as samples in the full countercurrent system, and tested again for moisture content. The correction factor was 0.12 g moisture per 100 g of solvent. Recovered solvent values were recalculated and termed "corrected recovered solvent moisture contents."

There were significant differences in the corrected moisture contents of the recovered solvent of all three treatments. Changing both the solvent and configuration of the system significantly decreased the moisture content of the recovered solvent (table 3). The moisture content of the corrected recovered solvent of the single–pass 30% hexanes was less than half that of the control.

Moisture levels of the miscellas of each stage at steady–state (table 4) show that the control had higher moisture levels in all stages than the single– or multi–pass 30% hexanes treatments. Most of the water was adsorbed in stages 1 and 2 because these are the first miscellas to come in contact with fresh dry corn. Little drying occurred in subsequent extraction stages, indicating that adding stages to improve solvent drying may be unwarranted. The single–pass 30% hexanes treatment gave the largest difference in moisture contents between the incoming solvent and full (oldest) miscella, which indicated more efficient adsorption, thus confirming the results of the breakthrough adsorption and extraction curves with regard to the effect of L/D ratio on water adsorption (Miller, 1995).

Oil yield was calculated by weighing the material remaining after evaporating the full miscella and dividing this weight by the average batch weight of dried corn. Oil yields for the single–pass and multi–pass 30% hexanes treatments were significantly greater than that of the control (table 5). The residual oil in the defatted corn was also significantly lower in both extractions that used 30% hexanes. The lower residual oil content was attributed to decreased polarity of the solvent and thus greater propensity for oil. A mixture of ethanol and hexanes would be expected to extract the oil more easily than just ethanol alone. Changing the configuration of the system also improved oil extraction. Oil recoveries increased by changing both solvent and extraction column geometry. An oil recovery rate of greater than 95% was achieved and left only 0.2% oil in the defatted corn.

Oil yields for both the single– and multi–pass 30% hexanes treatments were greater than the original oil content of the corn (table 5). The starting oil content of the corn was determined by hexane extraction, as specified by approved methods. Oil yield calculations, on the other hand, used oil that was extracted by a mixture of hexanes and ethanol, which should extract a broader range of lipid material. These results suggest that the quality of oil extracted by the new SEP conditions may be lower, thus requiring more refining than crude oil extracted by the original SEP system.

The full miscella had the highest oil contents, while the oil contents of the subsequent miscellas were lower (table 4), as

Table 4. Moisture, oil, and protein contents of each miscella at steady–state.[a]

	Moisture content (% wb)			Oil content (%)			Protein content (%)	
Miscella	Control[b]	SEP 1[c]	SEP 2[d]	Control	SEP 1	SEP 2	SEP 1	SEP 2
1 (full)	2.18 ±0.02	0.84 ±0.01	0.47 ±0.01	3.00 ±0.08	3.34 ±0.03	4.54 ±0.05	0.02 ±0.01	0.01 ±0.00
2	2.68 ±0.08	1.37 ±0.01	1.65 ±0.01	1.62 ±0.02	1.62 ±0.01	1.09 ±0.02	0.02 ±0.00	0.03 ±0.01
3	2.61 ±0.07	1.78 ±0.02	2.01 ±0.01	0.79 ±0.01	0.95 ±0.01	0.69 ±0.01	0.02 ±0.00	0.02 ±0.00
4	2.90 ±0.08	1.86 ±0.01	2.10 ±0.01	0.44 ±0.09	0.62 ±0.02	0.52 ±0.01	0.02 ±0.00	0.02 ±0.00
5	2.95 ±0.14	1.98 ±0.02	2.13 ±0.05	0.26 ±0.02	0.44 ±0.01	0.39 ±0.01	0.02 ±0.01	0.02 ±0.00
6	3.19 ±0.12	2.06 ±0.03	2.19 ±0.05	0.15 ±0.05	0.32 ±0.01	0.30 ±0.02	0.01 ±0.00	0.02 ±0.01
7	3.89 ±0.02	2.13 ±0.02	2.20 ±0.02	0.06 ±0.00	0.21 ±0.01	0.19 ±0.00	0.01 ±0.00	0.01 ±0.01
Incoming	3.51 ±0.18	2.11 ±0.06	2.16 ±0.01	0	0	0	0	0

[a] All values are as–is basis.
[b] Original SEP system (multi–pass, using aqueous ethanol at 76°C).
[c] Multi–pass SEP system using 30% hexane:70% ethanol at 56°C.
[d] Single–pass SEP system using 30% hexane:70% ethanol at 56°C and extraction column with L/D ratio of 15.

Table 5. Oil recoveries from corn during countercurrent extraction.

Parameters	Control[a]	Multi–pass system, 30% hexanes[b]	Single–pass system, 30% hexanes[b]
System change	None	Solvent	Solvent and column geometry
Oil yield (g oil/100 g corn)[c]	3.77 ±0.19 b	5.13 ±0.12 a	5.48 ±0.12 a
Starting oil content of corn (% db)	4.01 ±0.11	4.01 ±0.11	4.01 ±0.11
Residual oil in defatted corn (% db)[c]	0.30 ±0.08 a	0.25 ±0.02 b	0.18 ±0.04 c
Oil recovery (% db)[d]	90.8	93.8	95.5

[a] Means of 6 runs at steady–state.
[b] Means of 7 runs at steady–state.
[c] Means within a row followed by a common letter are not significantly different at $p < 0.05$.
[d] Based on residual oil contents of defatted corn.

would be expected. These concentrations demonstrate that most of the oil was extracted the first time the solvent went through the flake bed. Efficiency of oil extraction in the column with L/D ratio of 15 was much higher than the column with L/D ratio of 2.

Protein extraction was significantly affected by addition of PAS and by changing the extraction column geometry (table 6). Addition of PAS and lower extraction temperature reduced the amount of protein extracted from the corn by more than 50%. Changing the extraction column geometry effectively eliminated the extraction of protein. Protein levels of both miscellas were at the detection level.

The protein contents of the miscellas in each stage were almost undetectable (table 4). The concentrations of protein in the miscellas of each treatment were constant; there was no gradient, which indicated that length of extraction time had little or no effect on protein concentration.

CONCLUSION

The solvent mixture of 70% ethanol (4.5% moisture, wb):30% hexanes and extraction temperature of 56°C were the most effective conditions for minimizing protein co–extraction with oil while maintaining high values for oil recovery and moisture adsorption by corn. Greater water adsorption and more efficient oil and protein extractions protein, and water transferred were also greater. Extraction were achieved by using the column with a larger L/D ratio. Not only was the extraction faster, the total amounts of oil, was more efficient in the long, narrow column because the solvent was forced to encounter more flaked corn before exiting the column. Improved conditions for the oil extraction/water adsorption step of SEP were identified as a single–pass system using an extraction column with L/D ratio of 15, 30% hexanes:70% ethanol as solvent, and extraction temperature of 56°C. Oil recoveries of 95% were maintained, water content in the recovered solvent was reduced, and protein extraction was drastically reduced. These improvements will facilitate the development of the two–step protein extraction that will be tested in future work.

Table 6. Crude protein (CP) contents of corn during countercurrent extraction.

Parameters	Control[a]	Multi–pass system, 30% hexanes[b]	Single–pass system, 30% hexanes[b]
System change	None	Solvent	Solvent and column geometry
Starting CP of corn (% db)	8.36 ±0.11	8.36 ± 0.11	8.36 ±0.11
Residual CP in defatted corn (% db)[c]	6.72 ±0.13 c	7.83 ±0.17 b	8.35 ±0.45 a
CP extracted (% db)	19.5	6.3	0.1

[a] Means of 6 runs at steady–state.
[b] Means of 7 runs at steady–state.
[c] Means within a row followed by a common letter are not significantly different at $p < 0.05$.

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